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**Semiconductor devices – Micro-electromechanical devices –
Part 42: Measurement methods of electro-mechanical conversion characteristics
of piezoelectric MEMS cantilever**

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SEMICONDUCTOR DEVICES – MICRO-ELECTROMECHANICAL DEVICES –

Part 42: Measurement methods of electro-mechanical conversion characteristics of piezoelectric MEMS cantilever

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The text of this International Standard is based on the following documents:

Draft	Report on voting
47F/414/FDIS	47F/417/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/standardsdev/publications.

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SEMICONDUCTOR DEVICES – MICRO-ELECTROMECHANICAL DEVICES –

Part 42: Measurement methods of electro-mechanical conversion characteristics of piezoelectric MEMS cantilever

1 Scope

This part of IEC 62047 specifies measuring methods of electro-mechanical conversion characteristics of piezoelectric thin film on microcantilever, which is typical structure of actual micro sensors and micro actuators. In order to obtain actual and precise piezoelectric coefficient of the piezoelectric thin films with microdevice structures, and this document reports the schema to determine the characteristic parameters for consumer, industry or any other applications of piezoelectric devices. This document applies to piezoelectric thin films on microcantilever fabricated by MEMS process.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological database for use in standardization at the following addresses:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

3.1

unimorph microcantilever

micro-scale cantilever composed of piezoelectric thin film and non-piezoelectric material, typically thin silicon layer

Note 1 to entry: The piezoelectric thin films are deposited on bottom electrode. Top electrodes are prepared on the piezoelectric thin films and input voltage are applied between top and bottom electrodes. Platinum is often used as top and bottom electrodes for piezoelectric MEMS devices. The thickness of both top and bottom electrodes should be thinner than that of piezoelectric thin film and non-piezoelectric layer of microcantilever. In case of direct piezoelectric measurements, output signal is measured between bottom electrode and sensing top electrode as described in 5.4.

3.2

converse transverse piezoelectric coefficient

transverse piezoelectric coefficient of the piezoelectric thin film calculated from strain or stress caused by electric field or voltage

3.3

direct transverse piezoelectric coefficient

transverse piezoelectric coefficient of the piezoelectric thin film calculated from generated charge or voltage caused by strain or stress

4 Test bed of MEMS piezoelectric thin film

4.1 General

These measuring methods of the transverse piezoelectric properties apply to the unimorph microcantilevers.

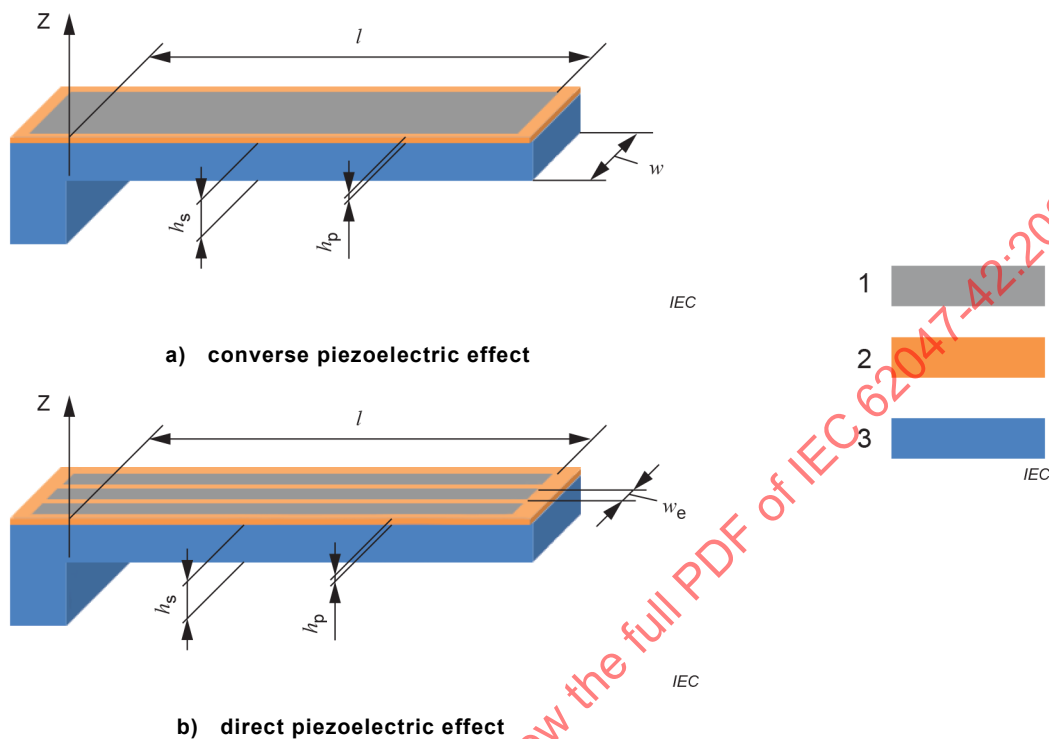


Figure 1 – Test bed of piezoelectric MEMS unimorph cantilever

Symbols and designations of test bed are given in Table 1.

Table 1 – Symbols and designations of test bed

Kind of properties	Symbol	Unit	Designation
Dimension of cantilever specimen	l	m	length of microcantilever
	w	m	width of microcantilever
	w_e	m	width of sensing top electrode
	h_s	m	thickness of non-piezoelectric layer
	h_p	m	thickness of piezoelectric thin film
Electro-mechanical conversion properties	$e_{31,f}$	C/m ²	effective transverse piezoelectric coefficient
	$e_{31,f}^d$	C/m ²	effective transverse piezoelectric coefficient (direct effect)
	$e_{31,f}^c$	N/Vm	effective transverse piezoelectric coefficient (converse effect)
	$e_{31,f}^c(V_{in,0})$	N/Vm	extrapolated effective transverse piezoelectric coefficient at 0V (converse effect)
	$e_{31,f}^c(V_{in,min})$	N/Vm	minimum effective transverse piezoelectric coefficient (converse effect at the lowest V_{in})
	$e_{31,f}^c(V_{in,max})$	N/Vm	maximum effective transverse piezoelectric coefficient (converse effect)
	d_{31}	m/V	transverse piezoelectric coefficient (d-form)
Electrical properties	C	F	capacitance between sensing top electrode and bottom electrode
	Q_{out}	C	output electric charge
	V_{in}	V	input peak-to-peak voltage
	$\tan \delta$		dielectric loss
	ω	rad/s	angular frequency
Mechanical properties	E	N/m ²	Young's modulus of microcantilever
	I	m ⁴	area moment of inertia of microcantilever
	ρ	kg/m ³	density of microcantilever
	D	m	tip displacement at x1
	E_s	N/m ²	Young's modulus of non-piezoelectric layer
	ν_s		Poisson's ratio of non-piezoelectric layer
	E_p	N/m ²	Young's modulus of piezoelectric thin film
	ν_p		Poisson's ratio of piezoelectric layer
	y_c	m	position of neutral plane of the unimorph cantilever from the bottom
	s_{11}^E, s_{12}^E	m ² /N	elastic compliances of piezoelectric thin film

4.2 Functional blocks and components

4.2.1 General

Figure 1 provides typical structure of the specimens consisted of piezoelectric unimorph microcantilever fabricated from a PZT (lead zirconate titanate ($\text{Pb}[\text{Zr}(x)\text{Ti}(1-x)]\text{O}_3$)) thin film on a Si (Silicon) or SOI (Silicon On Insulator) wafer. For the converse piezoelectric measurements, the surface of the unimorph microcantilever is covered by single top electrode where actuation voltage is applied (Figure 1 a)). Width of the top electrode is almost same as that of microcantilever. On the other hand, for direct piezoelectric measurements, top electrode is divided into three parts so that the bending vibration is generated by applying actuation voltage on two actuation electrodes, and simultaneously output voltage or charge by direct piezoelectric effect is measured from the centre sensing electrode (Figure 1 b)).

Details of the functional blocks or components named as the keys are provided in 4.2.2 to 4.2.4.

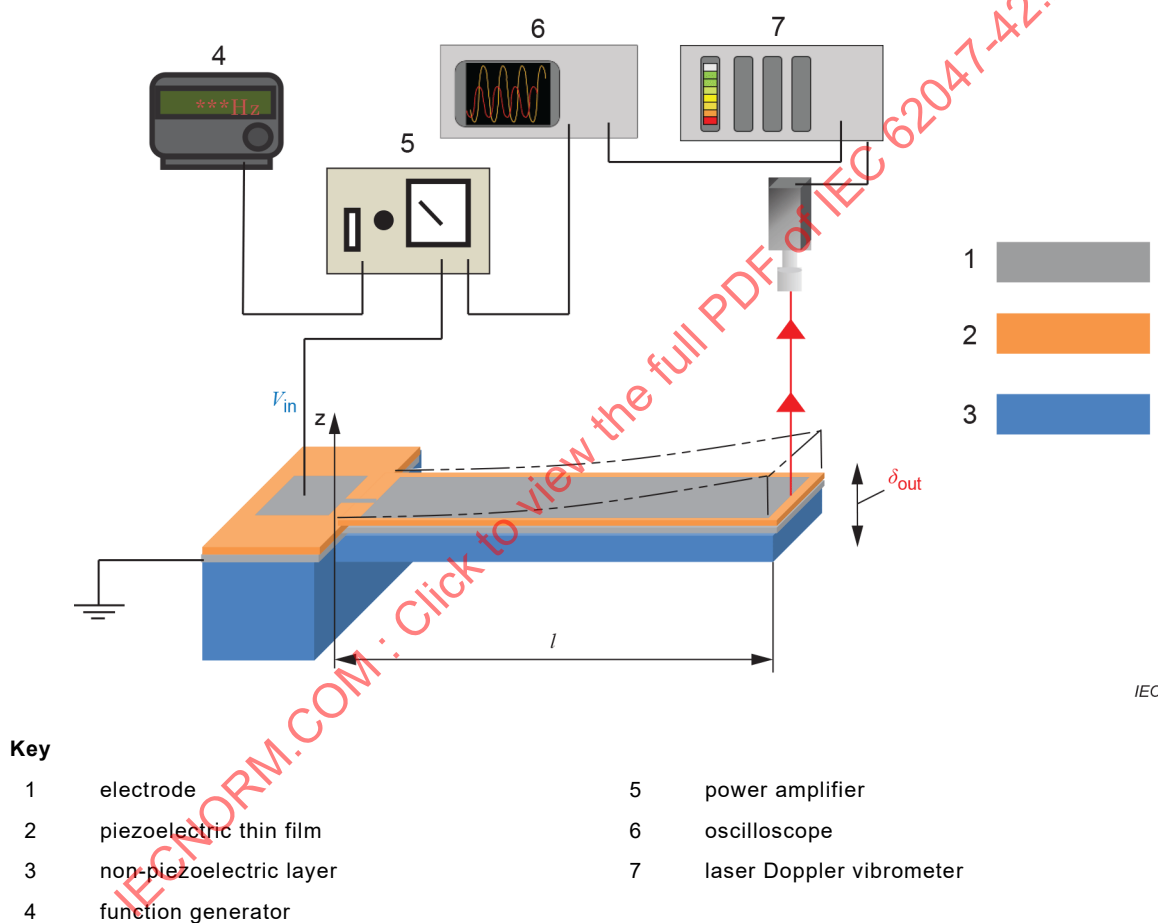
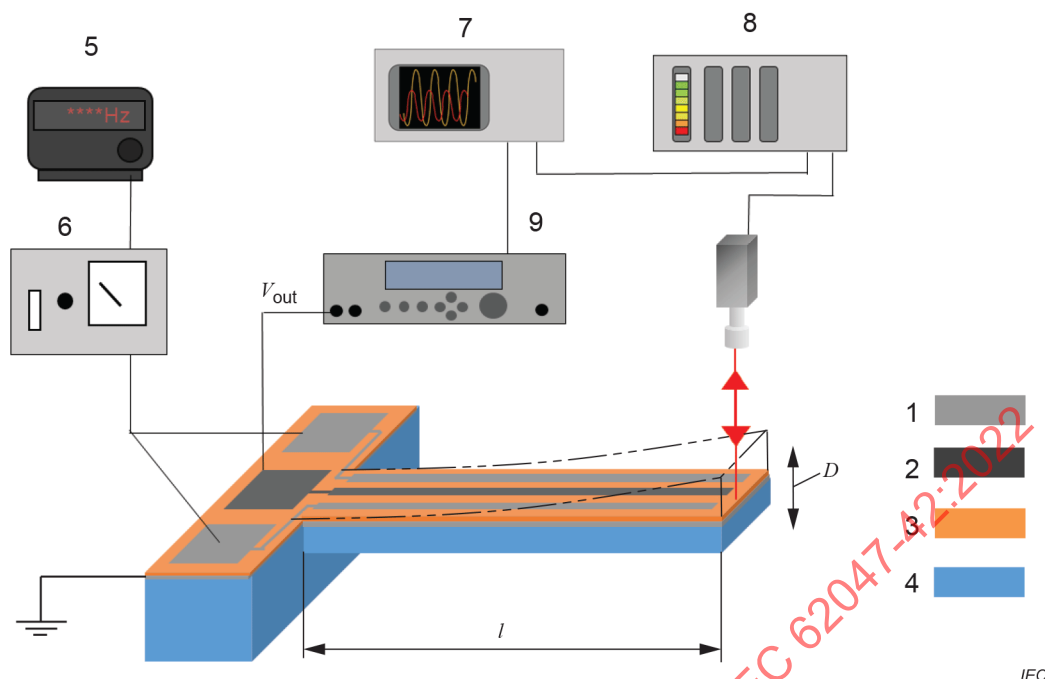


Figure 2 – Setup for measurement of converse piezoelectric effect



Key

- | | | | |
|---|-------------------------|---|--------------------------|
| 1 | actuation electrodes | 6 | power amplifier |
| 2 | sensing electrode | 7 | oscilloscope |
| 3 | piezoelectric thin film | 8 | laser Doppler vibrometer |
| 4 | non-piezoelectric layer | 9 | current amplifier |
| 5 | function generator | | |

Figure 3 – Setup for measurement of direct piezoelectric effect

4.2.2 Displacement meter

Displacement meter measures the tip displacement of the cantilever. Laser Doppler vibrometer can be used for this measurement.

4.2.3 Power source

In measurements of both converse and direct piezoelectric effects, actuation voltage for bending microcantilever is applied between top and bottom actuation electrodes by power source. Input sinusoidal signal from function generator is amplified by power amplifier.

4.2.4 Electric measurement instrument

In measurements of direct piezoelectric effect, the piezoelectric thin film of microcantilever under test generates electric output between sensing top and bottom electrodes by bending motion of microcantilever. Electric measurement instrument (i.e. voltmeter, charge meter, ammeter, current amplifier, oscilloscope, or lock-in amplifier) measures the generated voltage, charge or current synchronizing with the bending motion of the microcantilever generated by applying actuation voltage on the top electrodes.

5 Microcantilever under testing

5.1 General

Top surface of piezoelectric unimorph microcantilever is coated with single or three-separated top electrodes to measure converse and direct piezoelectric coefficients, respectively, as shown in Figure 1. As for converse piezoelectric effect, input voltage is applied on the single top electrode and output tip displacement is measured. On the other hand, as for direct piezoelectric effect, output voltage, charge or current is measured from the bending deformation of the unimorph microcantilever driven by actuation electrodes.

The thickness of the base material of the unimorph microcantilever shall be reduced, typically less than 50 µm, to release the internal stress of the piezoelectric thin films.

NOTE1 Generally, the thickness of the electrodes are much smaller than that of the piezoelectric thin film under testing.

NOTE2 In case of ferroelectric thin films, a poling treatment is usually done to align the polar direction and maximize their piezoelectric characteristics. Short-time application of larger voltage (AC or DC) than coercive voltage, which is determined by polarization-electric field hysteresis curves, is enough for the poling treatment of piezoelectric thin films.

5.2 Measurement principle

In general, effective transverse piezoelectric coefficient of piezoelectric thin film is defined as Formulas (1) to (4).

$$e_{31,f}^c = \frac{d_{31}}{s_{11}^E + s_{12}^E} \quad (1)$$

Effective transverse piezoelectric coefficient of converse piezoelectric effect ($e_{31,f}^c$) is calculated as follows.

$$e_{31,f}^c = -\frac{4D}{3V_{in}l(h_p + h_s)} \left[\frac{E_s \left\{ (h_s - y_c)^3 + y_c^3 \right\}}{1 - \nu_s} + \frac{E_p \left\{ (h_p + h_s - y_c)^3 - (h_s - y_c)^3 \right\}}{1 - \nu_p} + \frac{3h_p(h_p + h_s)}{2(s_{11}^E + s_{12}^E)} \left(h_s + \frac{h_p}{2} - y_c \right) \right] \quad (2)$$

Position of neutral plane y_c is calculated by the formula (3),

$$y_c = \frac{E_p h_p^2 + 2E_s h_p h_s + E_s h_s^2}{2(E_p h_p + E_s h_s)} \quad (3)$$

Effective transverse piezoelectric coefficient of direct piezoelectric effect ($e_{31,f}^d$) is calculated as follows. In this case, the frequency of input voltage shall be resonance frequency of mechanical vibration of microcantilever.

$$e_{31,f}^d = \frac{l(\sinh\lambda\cos\lambda - \sin\lambda\cosh\lambda)}{\lambda \cdot \sinh\lambda\sin\lambda} \frac{E_p h_p + E_s h_s}{E_s h_s(h_p + h_s)} \frac{Q_{out}}{D} \quad (4)$$

where λ of the first resonance is 1,875.

NOTE 1 Converse piezoelectric effect of some piezoelectric thin films shows strong dependence on the input voltage due to extrinsic piezoelectric contribution. Therefore, three transverse piezoelectric coefficients of $e_{31,f}^c(V_{in,min})$ and $e_{31,f}^c(V_{in,max})$ and $e_{31,f}^c(V_{in,0})$ are defined from the results of the converse piezoelectric measurements.

NOTE 2 $e_{31,f}^c(V_{in,max})$ is the lowest value of $e_{31,f}^c$, while $e_{31,f}^c(V_{in,min})$ is the highest value of $e_{31,f}^c$ in the measurements. Input voltage V_{in} of $e_{31,f}^c(V_{in,min})$ is lower than one-fifth of the input voltage V_{in} of $e_{31,f}^c(V_{in,max})$.

NOTE 3 $e_{31,f}^c(V_{in,0})$ is the value $e_{31,f}^c$ at input voltage $V_{in} = 0$. It can be obtained from the extrapolation curve of the relationship between $e_{31,f}^c$ and V_{in} . The equation of the extrapolation curve can be determined by the examiner, but it is indicated in the test report.

NOTE 4 Resonance frequency and Q factor is measured by frequency response of the displacement of the unimorph microcantilever. Q value can be determined by half-power bandwidth of the frequency response of the tip displacement.

NOTE 5 Relative dielectric constant and dielectric loss is measured by LCR meter or impedance analyser, typically at the frequency of 1 kHz.

NOTE 6 Because the elastic compliances of piezoelectric thin film, s_{11}^E and s_{12}^E , are difficult to be measured, those of bulk material from the literature can be used. In case of PZT, the elastic compliances s_{11}^E and s_{12}^E are typically 13.8×10^{-12} and -4.07×10^{-12} m²/N (Bibliography [6]¹), respectively. Poisson's ratio of piezoelectric thin film is also driven from these values.

5.3 Measuring procedures of converse transverse piezoelectric coefficient

Measurement setup is shown in Figure 2. Unimorph microcantilever under testing has single top electrode where the actuation voltage is applied. Following steps are measuring procedures:

- measure the ambient temperature and relative humidity;
- apply unipolar sinusoidal voltage to the unimorph microcantilever with single top electrode under testing to vibrate the cantilever. Application voltage is the same direction as poling treatment if poling treatment is done to the specimen. Frequency of the sinusoidal voltage shall be far lower than resonance frequency of microcantilever;
- measure input voltage to the piezoelectric thin film under testing;
- measure displacement of the tip of the cantilever;
- input voltage sweeps more than three times, and the final sweeping data are used for the evaluation of $e_{31,f}^c$.

5.4 Measuring procedures of direct transverse piezoelectric coefficient

Measurement setup is shown in Figure 3. Unimorph microcantilever under testing has three top electrodes. Two top electrodes except for centre are the actuation electrodes to generate resonance bending vibration of the microcantilever, while the centre top electrode is the sensing electrode to measure direct piezoelectric effect. Following steps are measuring procedures:

- measure the ambient temperature and relative humidity;
- apply unipolar sinusoidal voltage to the unimorph microcantilever with three top electrode under testing to vibrate the microcantilever. The frequency of input voltage shall be resonance frequency. Application voltage is the same direction as poling treatment if poling treatment is done to the specimen;
- measure displacement of the tip of the microcantilever;
- measure output voltage of the centre electrode.

¹ Numbers in square brackets refer to the Bibliography.

6 Test report

Test report shall include at least the following information:

- Mandatory
 - a) length, width, and thickness of the unimorph microcantilever
 - b) composition of the piezoelectric thin film under testing
 - c) material and its properties of piezoelectric and non-piezoelectric layer
 - Young's modulus, Poisson's ratio, thickness and crystal orientation in case of single crystal
 - d) electric properties of the piezoelectric thin film under testing
 - capacitance, relative dielectric constant and dielectric loss of sensing electrode
 - e) resonance frequency of microcantilever
 - f) test environment (temperature and relative humidity)
 - g) test conditions of converse transverse piezoelectric coefficient
 - Input voltage, waveform and operating non-resonance frequency applied to the microcantilever under testing
 - h) test conditions of direct transverse piezoelectric coefficient
 - Input voltage, waveform and operating resonance frequency of two actuation top electrodes, and tip displacement of microcantilever
 - i) poling treatment conditions
 - voltage
 - poling direction
 - temperature
 - waveform
 - poling time
 - time from the poling to the measurement
 - j) test items
 - $e_{31,f}^d$
 - $e_{31,f}^c(V_{in,min})$
 - $e_{31,f}^c(V_{in,max})$
- Optional
 - a) microfabrication process including deposition process of piezoelectric thin film
 - b) poling treatment conditions (opposite poling direction to the mandatory)
 - voltage
 - temperature
 - waveform
 - poling time
 - time from the poling to the measurement
 - c) test items (piezoelectric thin film treated by opposite poling direction)
 - $e_{31,f}^d$
 - $e_{31,f}^c(min)$
 - $e_{31,f}^c(max)$

d) extrapolated converse transverse piezoelectric coefficient at 0 V

- $e_{31,f}^c(0)$
- Q factor

An example of test results is described in Annex A.

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Annex A (informative)

Example of measuring method of piezoelectric MEMS cantilever

A.1 General

Annex A describes an example of measuring method of piezoelectric MEMS cantilever to obtain direct and/or converse piezoelectric coefficient of microfabricated piezoelectric thin film. Clauses A.2 to A.4 summarize the sample preparation procedures, poling treatment conditions, material properties for calculation of transverse piezoelectric coefficient, measuring procedures and measurement results.

A.2 Measurement procedure

A.2.1 Structure of piezoelectric microcantilevers

Piezoelectric microcantilevers are prepared by MEMS microfabrication process. Structure and picture of the piezoelectric microcantilevers under testing is shown in Figure A.1. Upper three microcantilevers have three separated top electrodes of actuating or sensing which are used for direct piezoelectric measurements, while lower three microcantilevers are used for converse piezoelectric measurements.

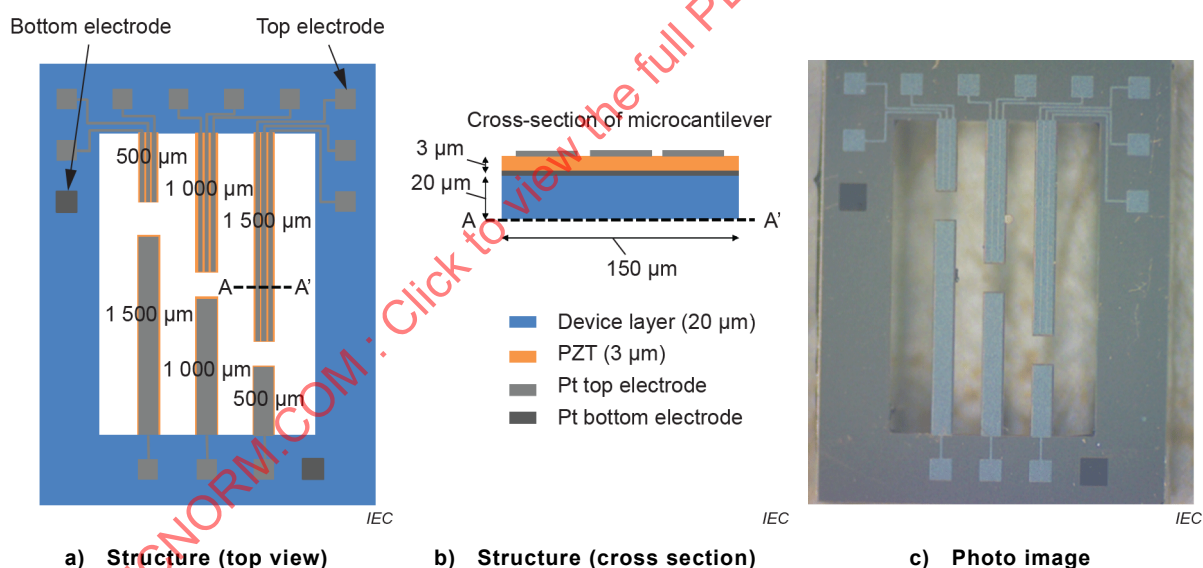
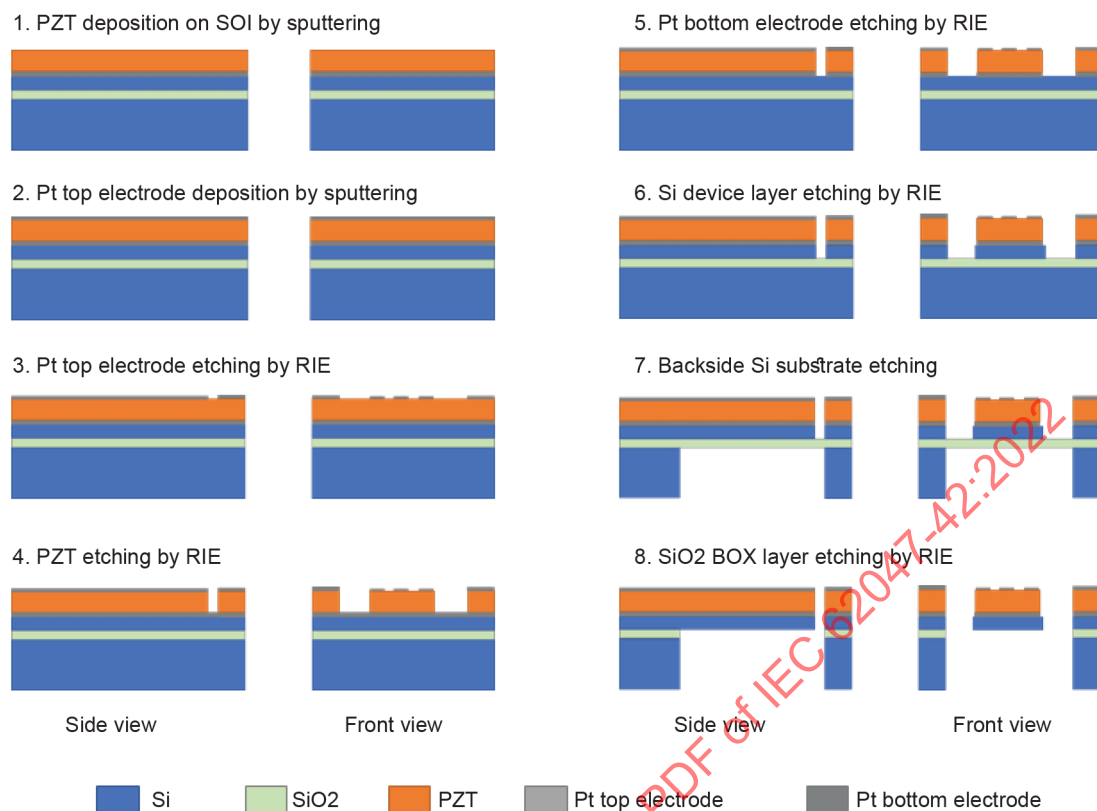


Figure A.1 – Structure and photograph of piezoelectric microcantilevers under testing

A.2.2 Microfabrication process

Microfabrication process of piezoelectric microcantilevers are shown in Figure A.2.



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Figure A.2 – Fabrication process of piezoelectric microcantilevers

A.2.3 Mechanical properties of piezoelectric and non-piezoelectric layers

Mechanical properties of each layer are used for the calculation of direct and converse piezoelectric coefficient $e_{31,f}$. Thickness and elastic properties of piezoelectric and non-piezoelectric layers are shown in Table A.1 and Table A.2, respectively.

Table A.1 – Mechanical properties of piezoelectric layer

Kind of properties	Mechanical properties
Material composition	Polycrystalline $\text{Pb}(\text{Zr}_{0,52}\text{Ti}_{0,48})\text{O}_3$
Thickness	3 μm
Elastic compliance s_{11}^E	$13,8 \times 10^{-12} \text{ m}^2/\text{N}$ [6]
Elastic compliance s_{12}^E	$-4,07 \times 10^{-12} \text{ m}^2/\text{N}$ [6]
Young's modulus E_p ($1/s_{11}^E$)	$72,5 \times 10^9 \text{ Pa}$
Poisson's ration ν_p ($-s_{12}^E/s_{11}^E$)	0,29

Table A.2 – Mechanical properties of non-piezoelectric layer

Kind of properties	Mechanical properties
Material	(100)Si single crystal
Crystal orientation of length	<110>
Thickness	20 µm
Young's modulus E_s	169×10^9 Pa [5]
Poisson's ratio ν_s	0,064 [5]

A.2.4 Electric properties and resonance frequency of microcantilever

For the calculation of direct piezoelectric coefficient $e_{31,f}^d$, capacitance of sensing electrode is needed for the calculation using Formula (4). Electric properties of piezoelectric layer are shown in Table A.3. Note that the capacitance includes the area of lead line and contact pad. Measurement is conducted at resonance and resonance frequency is needed for Formula (5). Resonance frequency of each microcantilever for direct piezoelectric measurements is shown in Table A.4. Frequency response of each microcantilever is shown in Figure A.3.

Table A.3 – Electric properties of microcantilever

Length of microcantilever	Kind of properties	Electric properties
500 µm	Capacitance	166,5 pF
	Relative dielectric constant	205
	Dielectric loss	3 %
1 mm	Capacitance	222,7 pF
	Relative dielectric constant	251
	Dielectric loss	3 %
1,5 mm	Capacitance	291 pF
	Relative dielectric constant	304
	Dielectric loss	3 %

Table A.4 – Resonance frequencies of microcantilever

Length of microcantilever	Resonance frequency
500 µm	102,2 kHz
1 mm	26 kHz
1,5 mm	11,7 kHz

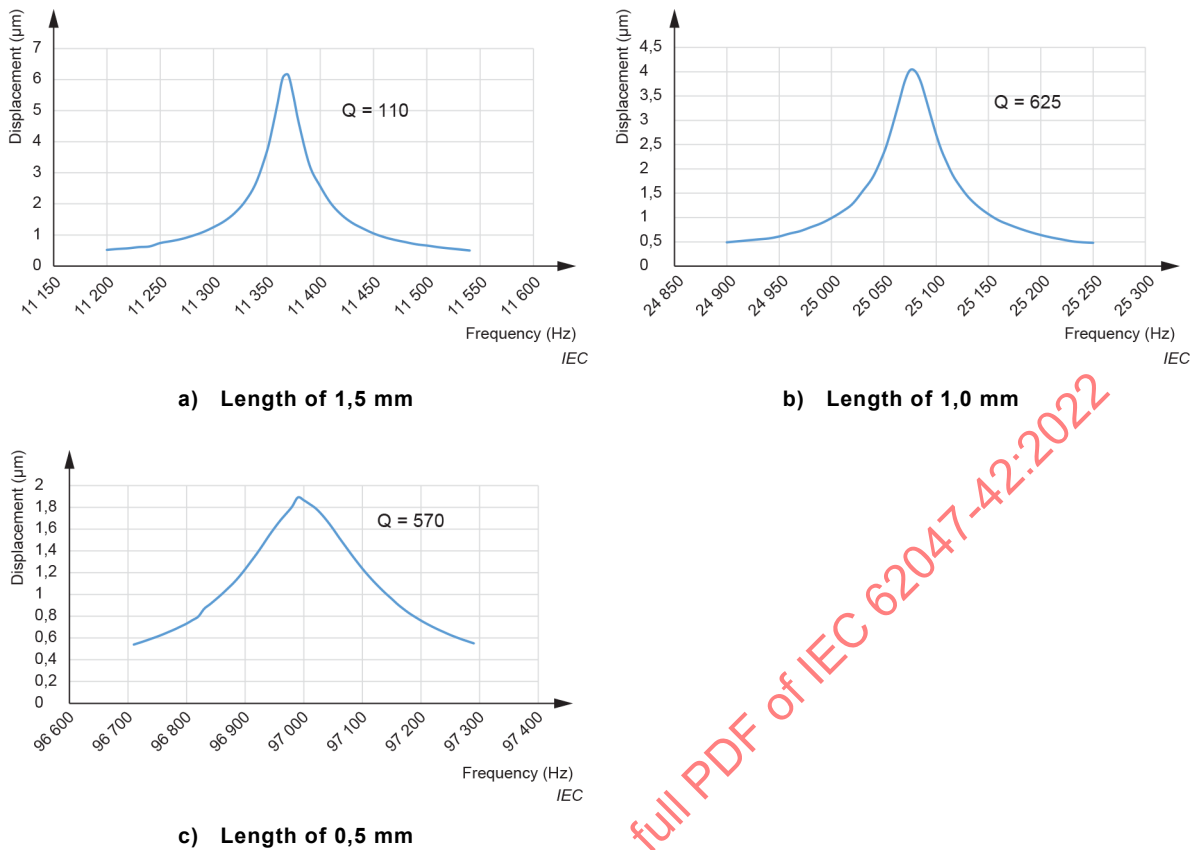


Figure A.3 – Frequency response of tip displacement of each piezoelectric microcantilevers

A.2.5 Input displacement of microcantilever for direct piezoelectric coefficient $e_{31,f}^d$

For direct piezoelectric measurements, mechanical stress or strain is applied to a piezoelectric layer. In case of the piezoelectric microcantilevers as shown in Figure A.1, actuating voltage is applied on two side actuating electrode and tip displacement is measured by laser Doppler vibrometer. Input signal is unipolar sine wave of the resonance vibration frequency with the same direction of electric field as polarization. Applied voltage, frequency and maximum tip displacement for direct piezoelectric measurements are shown in Table A.5.

Table A.5 – Input displacement for direct piezoelectric coefficient $e_{31,f}^d$

Length of microcantilever	Operation frequency (Resonance)	Maximum tip displacement
500 μm	102,2 kHz	2 μm
1 mm	26 kHz	4 μm
1,5 mm	11,7 kHz	6 μm